

UNITED STATES ENVIRONMENTAL PROTECTION AGENCY

REGION 4

Science and Ecosystem Support Division  
Ecological Support Branch

Standard Operating Procedure (SOP)  
For the Determination of Suspended Sediment Concentration (SSC)

Effective Date: 10/01/2001

SOP#: SEDXX

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Bruce A. Pruitt  
Author

\_\_\_\_\_  
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Date

Approvals

\_\_\_\_\_  
Work Assignment Manager

\_\_\_\_\_  
Date

\_\_\_\_\_  
Ecological Evaluation Section Chief

\_\_\_\_\_  
Date

\_\_\_\_\_  
Ecological Support Branch Quality Assurance Officer

\_\_\_\_\_  
Date

\_\_\_\_\_  
Ecological Support Branch Chief

\_\_\_\_\_  
Date

- For Documentation of Approval for Use in SESD, EAB
- Not for Distribution

## Procedure Section

### 1.0 Scope and Application

Substrate physical properties and fluvial sediment are among the most important factors in characterizing habitat suitability for aquatic macroinvertebrates and fish (Howard 1969, USEPA 1973, and Waters 1995). A direct relationship between habitat and biological diversity has been well established (USEPA 1978, Raven et al. 1998, and Pruitt and Howard 2000). The Ecological Assessment Branch (EAB) has developed and used the linkage between fluvial sediment, habitat, and biological diversity in both freshwater and marine ecosystems for over twenty-five years (Hicks et al. 1975, USEPA 1982, and Pruitt et al. 2001). Presently, EAB routinely characterizes substrate physical properties including embeddedness and sediment deposition as an integral part of the rapid bioassessment protocol (RBP, Barbour 1999) in riverine settings.

### 2.0 Summary of Method

This method is based on the *Standard Test Methods for Determining Sediment Concentration in Water Samples* (ASTM D 3977-97) and *Quality-Assurance Plan for the Analysis of Fluvial Sediment by the Northeastern Region, Kentucky District Sediment Laboratory* (USGS 1998). The method is based on measuring the total suspended sediment concentration (SSC) collected using environmental sampling techniques (see Fluvial Sediment Sampling). In contrast, to total suspended sediment (TSS), SSC is a measure of the total environmental sample rather than an aliquot as with TSS. Two methods are presented below: filtration and evaporation.

### 3.0 Interferences

Not applicable.

### 4.0 Health and Safety Procedures

Laboratory technicians should use precautions when working with convection ovens and muffle furnaces which operate at extremely high temperatures. The following safety equipment is recommended when removing samples from the convection oven and muffle furnace: faceshield, gloves, laboratory coats/aprons, and/or long tongs.

### 5.0 Special Procedures

Not applicable.

### 6.0 Analyst Training

Technicians/soil scientists are trained by experienced EPA personnel until proficient.

### 7.0 Reagents and Standards

Not applicable.

8.0 Apparatus and Materials

- 8.1 Polyethylene drop-dispensing bottles (Nalgene™ or equivalent).
- 8.2 Porcelain Gooch crucibles with fritted discs (appropriate size based on sample volume) (Coors™ or equivalent) or glass evaporation dishes or beakers.
- 8.3 Whatman #934-AH glass-fiber crucible filters.
- 8.3 Stainless steel utility trays.
- 8.4 Forced-air, gravity-convection oven (Fisher™ Isotemp or equivalent) equipped with a thermostat with range 90 to 120°C.
- 8.5 Top-loading, laboratory balance with a resolution of 0.0001 g and capacity of 150 g for weighing the dry sediments.
- 8.6 Top-loading, laboratory balance with a resolution of 0.1 g and a capacity of approximately 4000 g for weighing sample bottles containing water and sediment.
- 8.8 Desiccator cabinets.
- 8.9 Silica desiccant.
- 8.10 Graduated cylinder.
- 8.11 100 cc plastic syringe.
- 8.12 J-tube.
- 8.13 Filtration system with vacuum pump.

9.0 Sample Collection and Preservation

This SOP covers sample control and handling once the sample is received by the Sediment Characterization Laboratory (SCL) of EAB. Fluvial sediment is collected and transported to the laboratory at ambient air temperature. Once received by the SCL, sediment samples are maintained at room temperature, since SSC is a measure of inorganics. However, at the discretion of the project leader, samples can be preserved and maintained at 4°C.

10.0 Sample Holding Time

No maximum holding time is required.

11.0 Method Calibration

Not applicable

12.0 Sample Analysis and Procedure

12.1 Sample Preparation

Once samples are received, adequate time is allowed for settling. Settling time will be based on the particle size distribution of the sample. Determine the tare weight of all glassware and crucibles to be used during drying and weighing per instructions below.

12.2 Filtration Method

The following is a general step-wise procedure on the SSC using the filtration method. Modifications to the procedure should be requested through the SCL WAM prior to sample receipt. The filtration method can be used only on samples containing sand concentration less than approximately 10,000 ppm and clay concentrations less than 200 ppm. The sediment need not be settleable since filters are used to separate water from the sediment. Correction factors for dissolved solids are not required since the filtrate includes the dissolved solids, consequently it is not included in the final gravimetric weight.

- 12.2.1 Prior to receipt of environmental samples, wash the crucibles thoroughly, insert the filter, dry the crucible/filter pair at 105°C, cool to room temperature in the desiccator, determine the tare weight of the pair (crucible plus filter), and store the pair in the desiccator cabinet. Record the tare weight.
- 12.2.2 Immediately upon receipt of the environmental samples, mark the sample container at the water level. [Once the supernatant is withdrawn and the sediment/water mixture is decanted in the sieve, refill the sample container with de-ionized water to the mark and accurately measure the volume. Record the volume.]
- 12.2.3 Allow for adequate settling time.
- 12.2.4 Withdraw the supernatant using a J-tube being careful not to disturb the precipitate (approximately 40 to 70 mL of water-sediment mixture should be left). At the discretion of the project leader and SCL WAM, this step can be omitted if low sediment concentration is observed.
- 12.2.5 Separate the sands from the silt/clay fraction using a 62 µm sieve. Capture the silt/clay fraction in a suitable, tared glass beaker or evaporation disk. At the discretion of the project leader and SCL-WAM, the wet sieve separation of the silt/clay and sand fractions can be omitted if not required.
- 12.2.6 Decanter the sand and silt/clay fraction into separate crucible/filter pairs, wash the sample containers thoroughly into the crucible, and draw the water down by vacuum.
- 12.2.7 Place the crucible/filter pairs in the oven at 95°C until all visible traces of water have evaporated (over night is recommended). Then raise and hold the temperature at 105°C for approximately 2 hours.
- 12.2.8 Transfer the crucible/filter pairs to a desiccator, allow to cool to room temperature, and weigh to the nearest 0.0001 g as quickly as possible. Record the gross weight.

### 12.3 Evaporation Method

The following is a general step-wise procedure on the SSC using the evaporation method. Modifications to the procedure should be requested through the SCL WAM prior to sample receipt. This test is applicable to samples ranging from 0.2 to 20 L in volume, from 5 to 550,000 mg/L in sediment concentration, and having less than 35,000 mg/L in dissolved-solid concentration.

- 12.3.1 Prior to receipt of environmental samples, wash the beakers, crucibles or evaporation dishes thoroughly, dry the beaker at 105°C, cool to room temperature in the desiccator, determine the tare weight, and store the beaker in a desiccator cabinet. Record the tare weight.
- 12.3.2 Allow for adequate settling time.
- 12.3.3 Determine the dissolved-solids correction factor by transferring an aliquot (25 ml) into a glass beaker or evaporation dish.
- 12.3.4 Withdraw the supernatant using a J-tube being careful not to disturb the precipitate (approximately 40 to 70 mL of water-sediment mixture should be left).
- 12.3.5 Record the volume of the remaining water-sediment mixture for dissolved solids correction.

- 12.3.6 Decanter the remaining sediment/water mixture into the 62  $\mu\text{m}$  sieve, refill the sample container with de-ionized water to the mark and accurately measure the volume. Record the volume. At the discretion of the project leader and SCL-WAM, the wet sieve separation of the silt/clay and sand fractions can be omitted if not required.
- 12.3.7 Decanter the sand and silt/clay fraction into separate crucibles or evaporation dishes and wash the sample containers thoroughly into the crucible.
- 12.3.8 Evaporate the sediment/water mixtures (each fraction) and the dissolved solids aliquot from step 12.3.3 at 95°C to visible dryness (over night is recommended). Then raise and hold the temperature at 105°C for approximately 2 hours.
- 12.3.9 Transfer the beaker to a desiccator, allow to cool to room temperature, and weigh to the nearest 0.0001 g as quickly as possible. Record the weight of each fraction and the dissolved solids correction factor.

### 13.0 Quality Control

At the discretion of the project leader, quality control will be maintained with duplicate samples. In addition, sediment samples of known particle size distribution and weight may be utilized to determine loss of material during sieving.

### 14.0 Data Analysis and Calculation

- 14.1 Filtration method
  - 14.1.1 Net weight of coarse sediment ( $> 63 \mu\text{m}$ ) = gross weight of sediment/crucible - tare weight
  - 14.1.2 Net weight of fine sediment ( $< 63 \mu\text{m}$ ) = gross weight of sediment/crucible - tare weight
  - 14.1.3 Total weight sediment = net weight of coarse sediment + net weight of fine sediment
- 14.2 Evaporation method
  - 14.2.1 Net weight of coarse sediment ( $> 63 \mu\text{m}$ ) = gross weight of sediment/evaporation dish - tare weight
  - 14.2.2 Net weight of fine sediment ( $< 63 \mu\text{m}$ ) = gross weight of sediment/evaporation dish - tare weight
  - 14.2.3 Total weight sediment = net weight of coarse sediment + net weight of fine sediment
- 14.3 Dissolved solids correction
  - 14.3.1  $\text{DSc} = (\text{DS}/\text{Va}) \times \text{Vs}$   
where:  
DSc = dissolved solids correction, g  
DS = net weight of dissolved solids determined in 12.3.9, g  
Va = aliquot volume taken for dissolved solids in 11.7, mL, and  
Vs = volume of supernate remaining with the sediment from 12.3.6, mL.
  - 14.3.2 In the equation above, the DS/Va is the concentration of dissolved solids in the supernate. This concentration is multiplied by Vs to obtain the dissolved solids weight in the dry sediment. Record the value of Dsc.
  - 14.3.3 Subtract the value of DSc from the net weight of each fraction (steps 14.2.1 and 14.2.2) and the total weight of sediment (14.2.3).

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